## CLAIMS:

## What is claimed is:

- 1. A process for the recevery of acrylonitrile, methacrylonitrile or hydrogen cyanide obtained from the reactor effluent of an ammoxidation reaction of propane, propylene or isobutylene comprising passing said reactor effluent through an absorber column, a recovery column and a heads column wherein the improvement comprises operating said head column in a manner which inhibits the formation of an aqueous phase above the feed tray of said heads column.
- The process of claim 1, wherein said operating manner of said heads column
  comprises increasing the reflux ratio of said heads column to the point that no
  aqueous phase forms above the feed tray.
- The process of claim 2, wherein said operating manner of said heads column comprises feeding more hydrogen cyanide to said heads column to achieve conditions equivalent to higher reflux ratio.
- The process of claim 3, wherein said feeding results from recycling purified HCN to the heads column.

- The process of claim 3, wherein said feeding results from operating an ammoxidation reactor in a manner to produce said reactor effluent with high concentrations of HCN.
- The process of claim 1, wherein said operating manner of said heads column comprises using a side decanter to remove aqueous material from said heads column.
- The process of claim 1, wherein said operating manner of said heads column comprises increasing the number of stripping trays of said head column.
- The process of claim 1, wherein said operating manner of said heads column comprises increasing the reboiler duty of said heads column.
- 9. The process of claim 1, wherein said operating manner of said heads column comprises using an intermediate condenser above the feed tray of said heads column and below the reflux condenser of said heads column.
- 10. The process of claim 1, wherein said operating manner of said heads column comprises cooling the feed stream to said heads column to a temperature that no aqueous phase forms above the feed tray.

- 11. The process of claim 1, wherein said operating manner of said heads column comprises subcooling the reflux stream to said heads column.
- 12. The process of claim 1, wherein said operating manner of said heads column comprises reducing operating pressure of said heads column with or without other changes so that the second liquid phase region is reduced with pressure reduction as well.
- Acrylonitrile, methacrylonitrile or hydrogen cyanide produced using the process of claim 1.